

A Novel Microcharacterization Technique in the Measurement of Strain and Orientation Gradient in Advanced Materials

H. Garmestani^{1,2,3}, K. Harris³, L. Lourenco²

Center for Nonlinear & Nonequilibrium Aeroscience &
Florida A&M University
1800 E. Paul Dirac Rd., Tallahassee, FL 32306-4005

Department of Mechanical Engineering
FAMU-FSU College of Engineering
2525 Pottsdamer St., Tallahassee, FL 32310-2175

Center for Materials Research and Technology
Florida State University, Tallahassee, FL 32306-4000

Introduction

Representation of morphology and evolution of the microstructure during processing and their relation to properties requires proper experimental techniques. Residual strains, lattice distortion, and texture (micro-texture) at the interface and the matrix of a layered structure or a functionally gradient material and their variation are among parameters important in materials characterization but hard to measure with present experimental techniques. Currently techniques available to measure changes in interred material parameters (residual stress, micro-texture, microplasticity) produce results which are either qualitative or unreliable. This problem becomes even more complicated in the case of a temperature variation. These parameters affect many of the mechanical properties of advanced materials including stress-strain relation, ductility, creep, and fatigue. A review of some novel experimental techniques using recent advances in electron microscopy is presented here to measure internal stress, (micro)texture, interracial strength and (sub) grain formation and realignment. Two of these techniques are combined in the chamber of an Environmental Scanning Electron Microscope to measure strain and orientation gradients in advanced materials. These techniques which include Backscattered Kikuchi Diffractometry (BKD) and Microscopic Strain Field Analysis are used to characterize metallic and intermetallic matrix composites and superplastic materials. These techniques are compared with the more conventional x-ray diffraction and indentation techniques.

Experimental Techniques

EBSP and OIM Techniques

Electron backscattered pattern (EBSP) in the SEM originates from elastically backscattered and diffracted electrons. These electrons are formed when stationary primary electron beam is made to hit the surface of a specimen inclined at 70°. The diffraction patterns are imaged on a phosphor screen placed close to it, as illustrated in Figure 1. The phosphor screen is viewed through an optical port using a high gain television camera which in turn is interfaced to a computer. The resulting Kikuchi pattern (Figure 2) is recorded in the computer and indexed. By indexing successive patterns from hundreds of selected points on the sample surface, sufficient data can be collected to determine both macroscopic and

local orientation texture and to provide a detailed survey of nearest neighbor orientation relationships. Following upon the work of Venables[1], who developed the EBSP technique in the SEM, Dingley[2] advanced the technique, by interfacing with a computer to produce an on-line analysis of the diffraction patterns. This technique was later automated [3, 4] and evolved into the Orientation Imaging Microscopy (OIM) technique.

The OIM technique is essentially an extension of the electron backscattered patterns (EBSP) technique. Here, the EBSPs are collected from points on the sample surface over a regular grid and then automatically indexed.

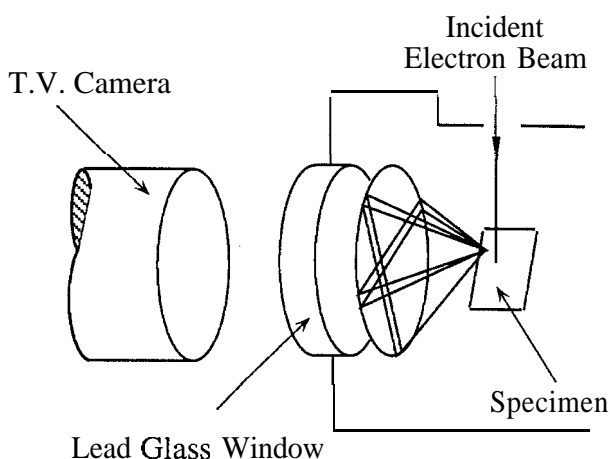


Figure 1- Schematics of BKD technique.



Figure 2- Typical Al-Kikuchi pattern

From this data, a map, called an OIM micrograph, is constructed displaying changes in crystal orientation over the specimen surface. In the OIM micrograph, the orientation of each point in the microstructure is known and hence the location, length and misorientation of all boundaries. This information is used to construct a micrograph based on a criteria input by the investigator. For example, a contiguous grain may be defined as a crystallographic entity on the basis that all points within it must have an orientation within 5°, 10°, or 15°. Typical OIM micrographs require several hundred to several thousand EBSP measurements to be taken on a hexagonal grid of points, with spacing of 0.2 mm to several microns.

In addition to the orientation measured, an image quality (IQ) parameter which represents the sharpness of the electron backscatter patterns can be determined. This parameter is associated with the presence and intensity of local plasticity and other defects. An IQ micrograph can be produced by mapping the data from each pixel over the entire region of interest. The distribution of grain boundary misorientations (mesotexture) and pole figures can also be constructed from the data. This fully automated technique provides for the examination of the microtexture and mesotexture of large regions of the specimen.

Microscopic Strain Field Analysis

We have developed a technique for microscopic strain field analysis (MSFA) which combines in situ heating or straining in an Environmental Scanning Electron Microscope (ESEM) with digital image processing [5]. This technique can be used to investigate the granular and intergranular strain localization during deformation. Figure 3 is a schematic which illustrates the application of MSFA to measure thermal strains in inhomogeneous materials. An ElectroScan model E-3 Environmental Scanning Electron Microscope

(ESEM) equipped with a heating and/or tensile stage, is used to image the specimen in a strain-free state and in a strained state. A digital micrograph of the region of interest is collected for each state. These images can be collected in real time (10 pictures per second) using a SGI computer interface. After the specimen is strained, the same feature must be centered before collecting the second micrograph.

The displacement field is determined by comparing the images of the strain-free and strained microstructure using the cross correlation approach. This approach has been discussed in detail for the measurement of particle displacements in the analysis of two dimensional velocity fields in fluid dynamics [6]. In brief, a small interrogation region where the material can be assumed to deform uniformly is selected. The displacement vector for the region is determined from the cross correlation of the equivalent regions in the two images. Two 2D Fourier transforms are applied to the interrogation region to obtain the two dimensional cross correlation function. In this two-dimensional array, the location of the maximum is proportional to the local displacement vector.

The cross correlation function is calculated for interrogation regions over the entire image to produce a map of the local displacement. Displacements are displayed as an array of vectors. The displacement gradients and the subsequent components of strain are calculated using a finite difference approach.

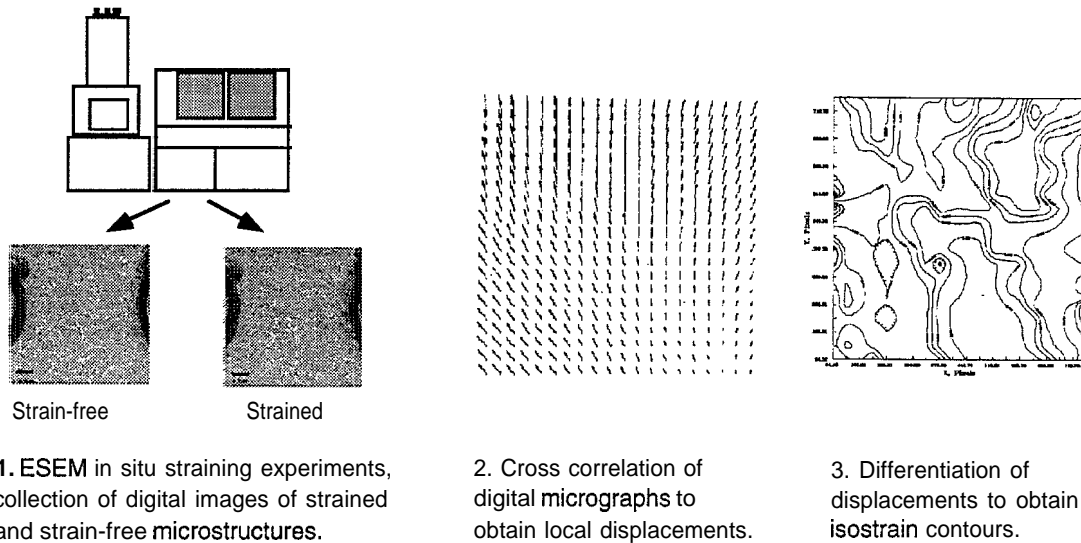


Figure 3- Schematic of MSFA for measurement of thermal strains in inhomogeneous materials.

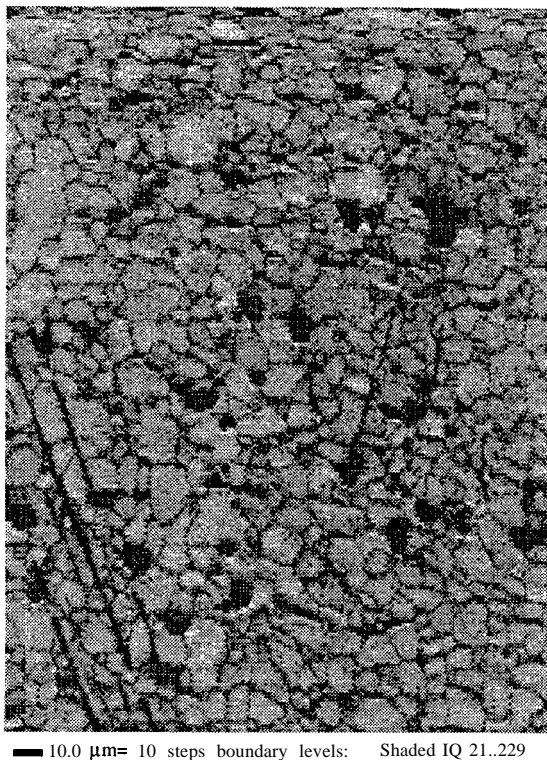
Results and Discussion

Materials and Materials development

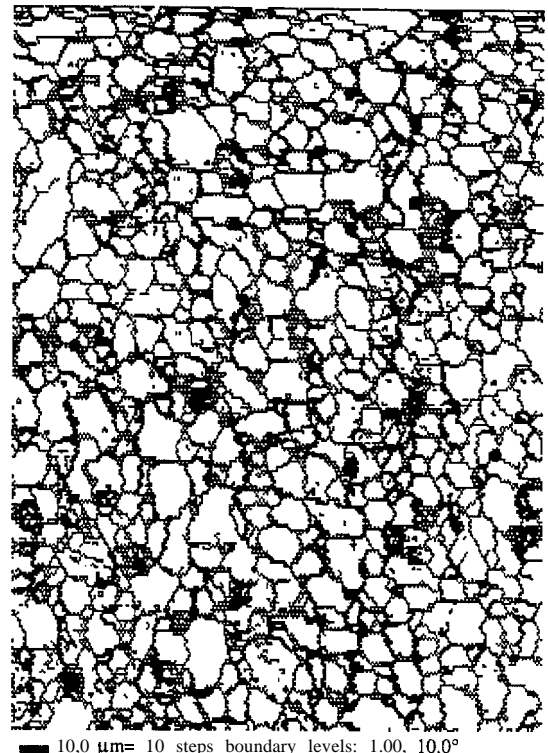
In this section, the preliminary results of micromechanical characterization of a Al-8090 superplastic material is presented. Tensile specimens of the material were deformed at 516 °C and at a rate of 10^{-4} sec⁻¹ to different strain values and then characterized. The techniques described above were used for characterization, and this include Orientation Imaging Microscopy (OIM), Electron Backscattered Kikuchi Pattern (EBSP), load relaxation and strain rate change tests.

OIM Microstructural Analysis

Figure 4-a is the IQ micrograph for a sample deformed to 15%. The microstructure is essentially equiaxed and similar to that obtained by conventional optical or scanning electron microscopy techniques. Figure 4-b is an OIM image constructed using the same data set as



(a)



(b)

Figure 4- a) Image Quality (IQ) micrograph and b) Disorientation boundary micrograph- Thin and thick lines represent grain boundaries with disorientation greater than 1° and greater than 10° respectively. Al-8090 specimen deformed to 15% strain.

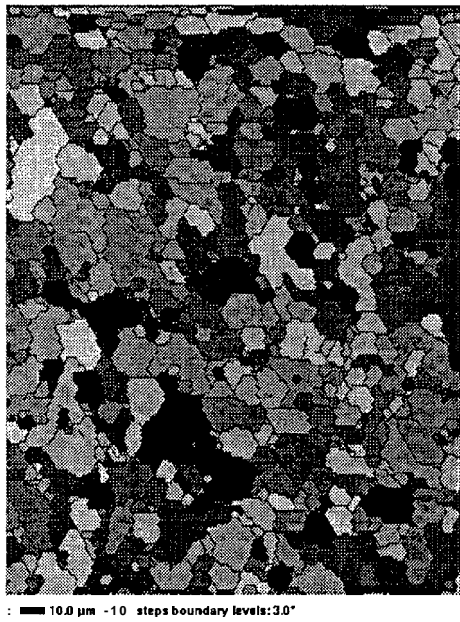


Figure 5- Disorientation boundary micrographs. Black lines depict boundaries with misorientations greater than 3° .

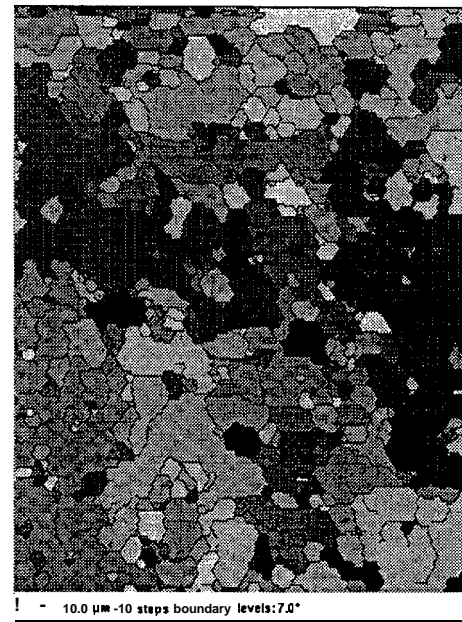


Figure 6- Misorientation boundary micrographs. Black lines depict boundaries with misorientations greater than 7° .

in Figure 6-a but drawn to reveal boundaries with disorientation between 1° and 10° as thin lines, and those boundaries with misorientation greater than 10° as thick lines. The microstructure appears to be equiaxed. If boundary disorientation greater than 3° is

chosen, the micrograph of Figure 5 is produced. In this micrograph, regions of the same orientation within a tolerance of 3° are distinguished by a uniform color. Similar analysis is presented in Figures 6 and 7-a for boundary disorientation greater than 7° , and 10° respectively. When boundaries with misorientations greater than 10° are plotted, the microstructure consists of fine grains sandwiched between coarse grain structures (Figure 7-a). The corresponding EBSD pole figures obtained from these grains are shown in Figure 7-b. It is clear that the microstructure exhibits a rotated cube type texture with three distinct pole segments, A, B and C corresponding to the three grains A, B and C in Figure 7-a.

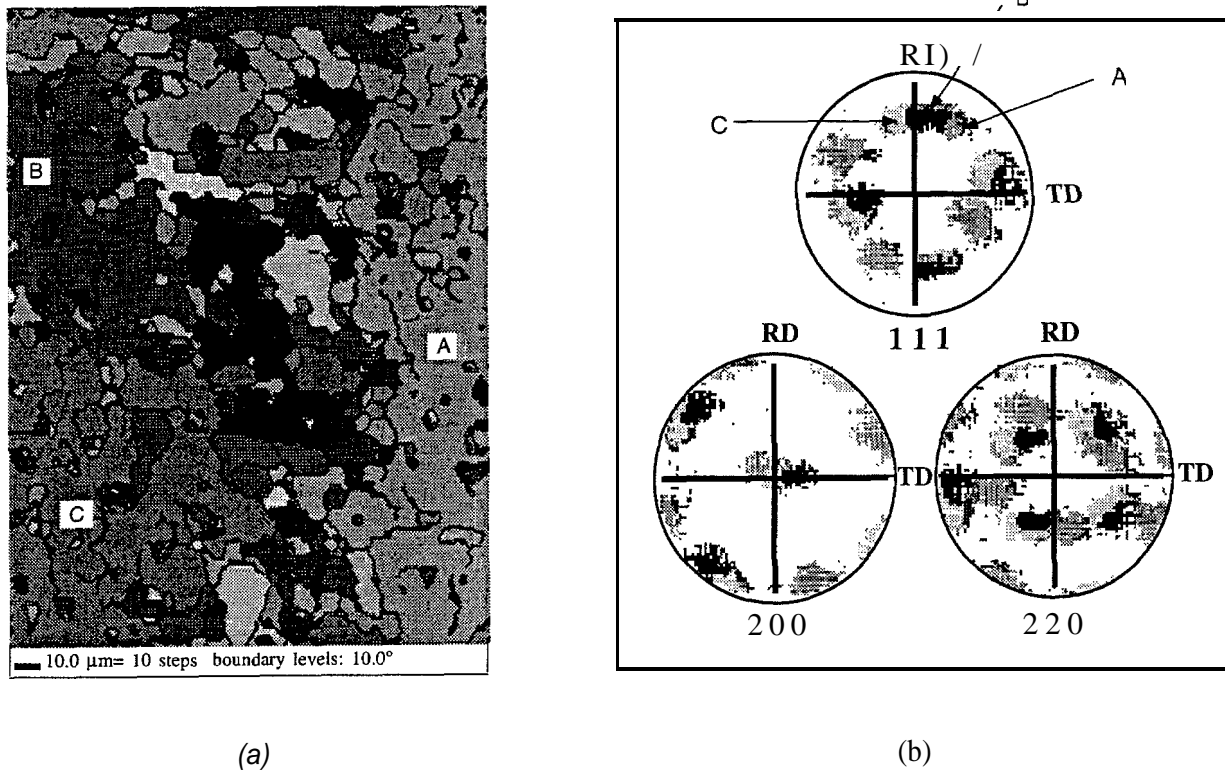


Figure 7- (a) 100 Disorientation boundary micrographs. (b) Pole figure representation of the previous figures. Dark gray, black and light gray represent the three right, top right and bottom left coarse regions.

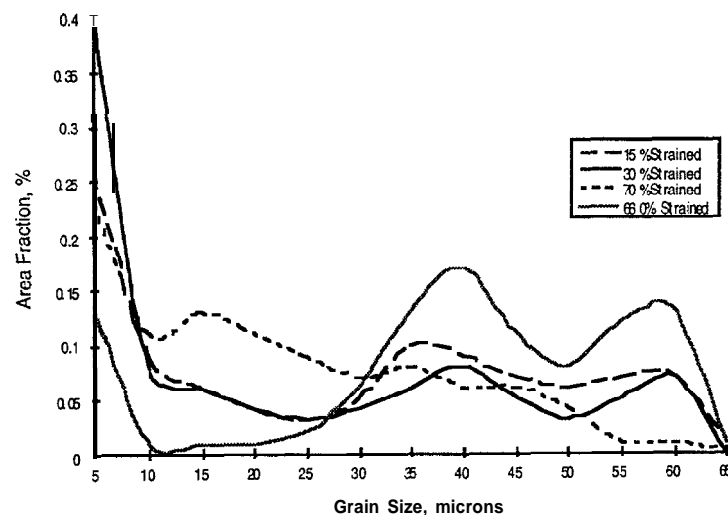


Figure 8- Composite of Test Results for Al-Li 8090 at (a) 516°C and (b) Disorientation histogram for AlLi-8090 at Four Strain Levels.

It is evident that these techniques have opened up a new horizon in the art of microstructural characterization. It is well accepted that grain boundary sliding occurs at high angle grain boundaries. The mechanisms of such process are still under argument. The experiments performed above can be analyzed to provide information on the nature of the grain boundaries and distinguish the high angle grain boundaries which are candidates for grain boundary sliding. Figure 8 provides the result of grain boundary disorientation distribution for different ranges of strain rates,

Summary and Conclusions

A novel microcharacterization technique is introduced which measures strain and texture gradients and uses the chamber of an Environmental Scanning Electron Microscope. It combines and adopts the most important features of two other techniques of characterization, EBSP and PIV (Particle Image Velocimetry). In general, this novel experimental technique will open new opportunities in the characterization of materials. It is shown that it was possible to measure strain and strain gradients with a resolution of 0.2% and orientation within 0.3 degrees. At the present these techniques are applied to superplastic materials and composites materials. It is intended to extend the range of applicability of these techniques to Nanostructures of high strain rate superplastic material using a Transmission Electron Microscope (TEM).

Acknowledgments

The funding for this work was provided by NASA through Cennas and Center for for Materials Research and Technology (MARTECH) and also through a grant by the Department of Transportation.

REFERENCES CITED

- [1] J. A. Venebles and C. J. Harland, in *Phil. Mag.*, vol. 27, 1973, pp. 1193-1200.
- [2] K. Z. Baba-Kishi and D. J. Dingley, "Backscatter Kikuchi Diffraction in the SEM for Identification of Crystallographic Point Groups," *Scanning*, vol. 11, pp. 305-312, 1989.
- [3] S. I. Wright, B. L. Adams, and K. Kunze, "Application of a new Automatic Lattice Orientation Measurement Technique to Polycrystalline Aluminum," *Materials Science and Engineering*, vol. A160, pp. 229-240, 1993.
- [4] H. Garmestani, P. N. Kalu, and D. Dingley, presented at Proceeding of the Light Weight Alloys for Aerospace Applications III, 124th Annual Meeting of TMS, 1995, Feb 12-16.
- [5] K. Harris, H. Garmestani, and L. Lourenco, "Microscopic Strain Field Analysis for In Situ ESEM Investigation," *to be submitted to the Journal of Experimental Mechanics*.
- [6] L. Lourenco and A. Krothapalli, "On the accuracy of velocity and vorticity measurements with PIV," *Experiments in Fluids*, vol. 18, pp. 421-428, 1995.